

## ROLL-OFF REDUCING AGENT

## BACKGROUND OF THE INVENTION

Field of the Invention

5        The present invention relates to a roll-off reducing agent. More specifically, the present invention relates to a roll-off reducing agent composition comprising the roll-off reducing agent, a process of reducing roll-off of a polished substrate by applying the roll-off reducing agent to the substrate, a process for producing a polished substrate by applying the roll-off reducing  
10      agent to the substrate, a polishing composition, a polishing process of a substrate to be polished by applying the polishing composition to the substrate, and a process for producing a substrate by applying the polishing composition to a substrate to be polished.

Discussion of the Related Art

Over the years, a demand for a technique for further increasing the capacity of the hard discs has become greater. As a leading means for increasing the capacity of the hard discs, there has been considered a means of producing a substrate capable of recording data even to its outer periphery by reducing roll-off (edge rounding of end side of a substrate) caused in the polishing process.  
20      For instance, various mechanical polishing conditions which can reduce roll-off, such as making a polishing pad more rigid, and making a polishing load smaller, have been studied. However, although a certain extent of an effect is obtained for reducing roll-off by these mechanical polishing conditions, the effect has not  
25      yet been satisfactory.

035242961590242475

In addition, as a polishing composition capable of reducing roll-off, a composition comprising water,  $\alpha$ -alumina particles and aluminum nitrate has been known (Japanese Patent Laid-Open No. Hei 9-286975). However, this composition does not have a satisfactory effect of reducing roll-off, and studies of the polishing components having an excellent effect for reducing roll-off have not yet been sufficiently made at present.

On the other hand, the hard discs have been greatly developed over the years in the trends of miniaturization and high capacity, so that the trend of high density has been progressed, that the minimum recording area has become smaller, and that the floating amount of a magnetic head has been made increasingly smaller. Therefore, there have been desired to increase the polishing rate and reduce the surface roughness, and to reduce surface defects such as scratches and pits in the polishing process of a hard disc substrate. In view of this, there have been studied on a polishing composition by using water, alumina, boehmite and a chelating compound (Japanese Patent Laid-Open No. Hei 11-92749, and the like); a polishing composition comprising water,  $\alpha$ -alumina, and an alumina sol stabilized with acetic acid (Japanese Patent Laid-Open No. 2000-63805); a polishing composition for an aluminum magnetic disc, comprising water, an alumina abrasive powder, a polishing accelerator such as gluconic acid or lactic acid, and a surface-modifying agent such as a colloidal alumina (Japanese Patent Laid-Open No. Hei 2-84485); a polishing composition comprising water, an alumina abrasive, and a polishing accelerator comprising molybdic acid and an organic acid (Japanese Patent Laid-Open No. Hei 7-216345); and a polishing process.

However, none of these polishing compositions sufficiently satisfy all of

10  
15  
TO/2410-69224860  
20

25

an effect of reducing roll-off, an effect of increasing the polishing rate, an effect of reducing the surface roughness of a polished object, and an effect of reducing surface defects such as scratches and pits. Therefore, a polishing composition having further enhanced effects is in demand.

5

An object of the present invention is to provide a roll-off reducing agent capable of reducing roll-off of a polished substrate caused by polishing, and increasing a polishing rate; a roll-off reducing agent composition comprising the roll-off reducing agent; a process of reducing roll-off of a polished substrate by using the roll-off reducing agent; and a process for producing a polished substrate by using the roll-off reducing agent.

010  
015  
020  
025  
030  
035  
040  
045  
050  
055  
060  
065  
070  
075  
080  
085  
090  
095  
100  
105  
110  
115  
120  
125  
130  
135  
140  
145  
150  
155  
160  
165  
170  
175  
180  
185  
190  
195  
200  
205  
210  
215  
220  
225  
230  
235  
240  
245  
250  
255  
260  
265  
270  
275  
280  
285  
290  
295  
300  
305  
310  
315  
320  
325  
330  
335  
340  
345  
350  
355  
360  
365  
370  
375  
380  
385  
390  
395  
400  
405  
410  
415  
420  
425  
430  
435  
440  
445  
450  
455  
460  
465  
470  
475  
480  
485  
490  
495  
500  
505  
510  
515  
520  
525  
530  
535  
540  
545  
550  
555  
560  
565  
570  
575  
580  
585  
590  
595  
600  
605  
610  
615  
620  
625  
630  
635  
640  
645  
650  
655  
660  
665  
670  
675  
680  
685  
690  
695  
700  
705  
710  
715  
720  
725  
730  
735  
740  
745  
750  
755  
760  
765  
770  
775  
780  
785  
790  
795  
800  
805  
810  
815  
820  
825  
830  
835  
840  
845  
850  
855  
860  
865  
870  
875  
880  
885  
890  
895  
900  
905  
910  
915  
920  
925  
930  
935  
940  
945  
950  
955  
960  
965  
970  
975  
980  
985  
990  
995  
1000  
1005  
1010  
1015  
1020  
1025  
1030  
1035  
1040  
1045  
1050  
1055  
1060  
1065  
1070  
1075  
1080  
1085  
1090  
1095  
1100  
1105  
1110  
1115  
1120  
1125  
1130  
1135  
1140  
1145  
1150  
1155  
1160  
1165  
1170  
1175  
1180  
1185  
1190  
1195  
1200  
1205  
1210  
1215  
1220  
1225  
1230  
1235  
1240  
1245  
1250  
1255  
1260  
1265  
1270  
1275  
1280  
1285  
1290  
1295  
1300  
1305  
1310  
1315  
1320  
1325  
1330  
1335  
1340  
1345  
1350  
1355  
1360  
1365  
1370  
1375  
1380  
1385  
1390  
1395  
1400  
1405  
1410  
1415  
1420  
1425  
1430  
1435  
1440  
1445  
1450  
1455  
1460  
1465  
1470  
1475  
1480  
1485  
1490  
1495  
1500  
1505  
1510  
1515  
1520  
1525  
1530  
1535  
1540  
1545  
1550  
1555  
1560  
1565  
1570  
1575  
1580  
1585  
1590  
1595  
1600  
1605  
1610  
1615  
1620  
1625  
1630  
1635  
1640  
1645  
1650  
1655  
1660  
1665  
1670  
1675  
1680  
1685  
1690  
1695  
1700  
1705  
1710  
1715  
1720  
1725  
1730  
1735  
1740  
1745  
1750  
1755  
1760  
1765  
1770  
1775  
1780  
1785  
1790  
1795  
1800  
1805  
1810  
1815  
1820  
1825  
1830  
1835  
1840  
1845  
1850  
1855  
1860  
1865  
1870  
1875  
1880  
1885  
1890  
1895  
1900  
1905  
1910  
1915  
1920  
1925  
1930  
1935  
1940  
1945  
1950  
1955  
1960  
1965  
1970  
1975  
1980  
1985  
1990  
1995  
2000  
2005  
2010  
2015  
2020  
2025  
2030  
2035  
2040  
2045  
2050  
2055  
2060  
2065  
2070  
2075  
2080  
2085  
2090  
2095  
2100  
2105  
2110  
2115  
2120  
2125  
2130  
2135  
2140  
2145  
2150  
2155  
2160  
2165  
2170  
2175  
2180  
2185  
2190  
2195  
2200  
2205  
2210  
2215  
2220  
2225  
2230  
2235  
2240  
2245  
2250  
2255  
2260  
2265  
2270  
2275  
2280  
2285  
2290  
2295  
2300  
2305  
2310  
2315  
2320  
2325  
2330  
2335  
2340  
2345  
2350  
2355  
2360  
2365  
2370  
2375  
2380  
2385  
2390  
2395  
2400  
2405  
2410  
2415  
2420  
2425  
2430  
2435  
2440  
2445  
2450  
2455  
2460  
2465  
2470  
2475  
2480  
2485  
2490  
2495  
2500  
2505  
2510  
2515  
2520  
2525  
2530  
2535  
2540  
2545  
2550  
2555  
2560  
2565  
2570  
2575  
2580  
2585  
2590  
2595  
2600  
2605  
2610  
2615  
2620  
2625  
2630  
2635  
2640  
2645  
2650  
2655  
2660  
2665  
2670  
2675  
2680  
2685  
2690  
2695  
2700  
2705  
2710  
2715  
2720  
2725  
2730  
2735  
2740  
2745  
2750  
2755  
2760  
2765  
2770  
2775  
2780  
2785  
2790  
2795  
2800  
2805  
2810  
2815  
2820  
2825  
2830  
2835  
2840  
2845  
2850  
2855  
2860  
2865  
2870  
2875  
2880  
2885  
2890  
2895  
2900  
2905  
2910  
2915  
2920  
2925  
2930  
2935  
2940  
2945  
2950  
2955  
2960  
2965  
2970  
2975  
2980  
2985  
2990  
2995  
3000  
3005  
3010  
3015  
3020  
3025  
3030  
3035  
3040  
3045  
3050  
3055  
3060  
3065  
3070  
3075  
3080  
3085  
3090  
3095  
3100  
3105  
3110  
3115  
3120  
3125  
3130  
3135  
3140  
3145  
3150  
3155  
3160  
3165  
3170  
3175  
3180  
3185  
3190  
3195  
3200  
3205  
3210  
3215  
3220  
3225  
3230  
3235  
3240  
3245  
3250  
3255  
3260  
3265  
3270  
3275  
3280  
3285  
3290  
3295  
3300  
3305  
3310  
3315  
3320  
3325  
3330  
3335  
3340  
3345  
3350  
3355  
3360  
3365  
3370  
3375  
3380  
3385  
3390  
3395  
3400  
3405  
3410  
3415  
3420  
3425  
3430  
3435  
3440  
3445  
3450  
3455  
3460  
3465  
3470  
3475  
3480  
3485  
3490  
3495  
3500  
3505  
3510  
3515  
3520  
3525  
3530  
3535  
3540  
3545  
3550  
3555  
3560  
3565  
3570  
3575  
3580  
3585  
3590  
3595  
3600  
3605  
3610  
3615  
3620  
3625  
3630  
3635  
3640  
3645  
3650  
3655  
3660  
3665  
3670  
3675  
3680  
3685  
3690  
3695  
3700  
3705  
3710  
3715  
3720  
3725  
3730  
3735  
3740  
3745  
3750  
3755  
3760  
3765  
3770  
3775  
3780  
3785  
3790  
3795  
3800  
3805  
3810  
3815  
3820  
3825  
3830  
3835  
3840  
3845  
3850  
3855  
3860  
3865  
3870  
3875  
3880  
3885  
3890  
3895  
3900  
3905  
3910  
3915  
3920  
3925  
3930  
3935  
3940  
3945  
3950  
3955  
3960  
3965  
3970  
3975  
3980  
3985  
3990  
3995  
4000  
4005  
4010  
4015  
4020  
4025  
4030  
4035  
4040  
4045  
4050  
4055  
4060  
4065  
4070  
4075  
4080  
4085  
4090  
4095  
4100  
4105  
4110  
4115  
4120  
4125  
4130  
4135  
4140  
4145  
4150  
4155  
4160  
4165  
4170  
4175  
4180  
4185  
4190  
4195  
4200  
4205  
4210  
4215  
4220  
4225  
4230  
4235  
4240  
4245  
4250  
4255  
4260  
4265  
4270  
4275  
4280  
4285  
4290  
4295  
4300  
4305  
4310  
4315  
4320  
4325  
4330  
4335  
4340  
4345  
4350  
4355  
4360  
4365  
4370  
4375  
4380  
4385  
4390  
4395  
4400  
4405  
4410  
4415  
4420  
4425  
4430  
4435  
4440  
4445  
4450  
4455  
4460  
4465  
4470  
4475  
4480  
4485  
4490  
4495  
4500  
4505  
4510  
4515  
4520  
4525  
4530  
4535  
4540  
4545  
4550  
4555  
4560  
4565  
4570  
4575  
4580  
4585  
4590  
4595  
4600  
4605  
4610  
4615  
4620  
4625  
4630  
4635  
4640  
4645  
4650  
4655  
4660  
4665  
4670  
4675  
4680  
4685  
4690  
4695  
4700  
4705  
4710  
4715  
4720  
4725  
4730  
4735  
4740  
4745  
4750  
4755  
4760  
4765  
4770  
4775  
4780  
4785  
4790  
4795  
4800  
4805  
4810  
4815  
4820  
4825  
4830  
4835  
4840  
4845  
4850  
4855  
4860  
4865  
4870  
4875  
4880  
4885  
4890  
4895  
4900  
4905  
4910  
4915  
4920  
4925  
4930  
4935  
4940  
4945  
4950  
4955  
4960  
4965  
4970  
4975  
4980  
4985  
4990  
4995  
5000  
5005  
5010  
5015  
5020  
5025  
5030  
5035  
5040  
5045  
5050  
5055  
5060  
5065  
5070  
5075  
5080  
5085  
5090  
5095  
5100  
5105  
5110  
5115  
5120  
5125  
5130  
5135  
5140  
5145  
5150  
5155  
5160  
5165  
5170  
5175  
5180  
5185  
5190  
5195  
5200  
5205  
5210  
5215  
5220  
5225  
5230  
5235  
5240  
5245  
5250  
5255  
5260  
5265  
5270  
5275  
5280  
5285  
5290  
5295  
5300  
5305  
5310  
5315  
5320  
5325  
5330  
5335  
5340  
5345  
5350  
5355  
5360  
5365  
5370  
5375  
5380  
5385  
5390  
5395  
5400  
5405  
5410  
5415  
5420  
5425  
5430  
5435  
5440  
5445  
5450  
5455  
5460  
5465  
5470  
5475  
5480  
5485  
5490  
5495  
5500  
5505  
5510  
5515  
5520  
5525  
5530  
5535  
5540  
5545  
5550  
5555  
5560  
5565  
5570  
5575  
5580  
5585  
5590  
5595  
5600  
5605  
5610  
5615  
5620  
5625  
5630  
5635  
5640  
5645  
5650  
5655  
5660  
5665  
5670  
5675  
5680  
5685  
5690  
5695  
5700  
5705  
5710  
5715  
5720  
5725  
5730  
5735  
5740  
5745  
5750  
5755  
5760  
5765  
5770  
5775  
5780  
5785  
5790  
5795  
5800  
5805  
5810  
5815  
5820  
5825  
5830  
5835  
5840  
5845  
5850  
5855  
5860  
5865  
5870  
5875  
5880  
5885  
5890  
5895  
5900  
5905  
5910  
5915  
5920  
5925  
5930  
5935  
5940  
5945  
5950  
5955  
5960  
5965  
5970  
5975  
5980  
5985  
5990  
5995  
6000  
6005  
6010  
6015  
6020  
6025  
6030  
6035  
6040  
6045  
6050  
6055  
6060  
6065  
6070  
6075  
6080  
6085  
6090  
6095  
6100  
6105  
6110  
6115  
6120  
6125  
6130  
6135  
6140  
6145  
6150  
6155  
6160  
6165  
6170  
6175  
6180  
6185  
6190  
6195  
6200  
6205  
6210  
6215  
6220  
6225  
6230  
6235  
6240  
6245  
6250  
6255  
6260  
6265  
6270  
6275  
6280  
6285  
6290  
6295  
6300  
6305  
6310  
6315  
6320  
6325  
6330  
6335  
6340  
6345  
6350  
6355  
6360  
6365  
6370  
6375  
6380  
6385  
6390  
6395  
6400  
6405  
6410  
6415  
6420  
6425  
6430  
6435  
6440  
6445  
6450  
6455  
6460  
6465  
6470  
6475  
6480  
6485  
6490  
6495  
6500  
6505  
6510  
6515  
6520  
6525  
6530  
6535  
6540  
6545  
6550  
6555  
6560  
6565  
6570  
6575  
6580  
6585  
6590  
6595  
6600  
6605  
6610  
6615  
6620  
6625  
6630  
6635  
6640  
6645  
6650  
6655  
6660  
6665  
6670  
6675  
6680  
6685  
6690  
6695  
6700  
6705  
6710  
6715  
6720  
6725  
6730  
6735  
6740  
6745  
6750  
6755  
6760  
6765  
6770  
6775  
6780  
6785  
6790  
6795  
6800  
6805  
6810  
6815  
6820  
6825  
6830  
6835  
6840  
6845  
6850  
6855  
6860  
6865  
6870  
6875  
6880  
6885  
6890  
6895  
6900  
6905  
6910  
6915  
6920  
6925  
6930  
6935  
6940  
6945  
6950  
6955  
6960  
6965  
6970  
6975  
6980  
6985  
6990  
6995  
7000  
7005  
7010  
7015  
7020  
7025  
7030  
7035  
7040  
7045  
7050  
7055  
7060  
7065  
7070  
7075  
7080  
7085  
7090  
7095  
7100  
7105  
7110  
7115  
7120  
7125  
7130  
7135  
7140  
7145  
7150  
7155  
7160  
7165  
7170  
7175  
7180  
7185  
7190  
7195  
7200  
7205  
7210  
7215  
7220  
7225  
7230  
7235  
7240  
7245  
7250  
7255  
7260  
7265  
7270  
7275  
7280  
7285  
7290  
7295  
7300  
7305  
7310  
7315  
7320  
7325  
7330  
7335  
7340  
7345  
7350  
7355  
7360  
7365  
7370  
7375  
7380  
7385  
7390  
7395  
7400  
7405  
7410  
7415  
7420  
7425  
7430  
7435  
7440  
7445  
7450  
7455  
7460  
7465  
7470  
7475  
7480  
7485  
7490  
7495  
7500  
7505  
7510  
7515  
7520  
7525  
7530  
7535  
7540  
7545  
7550  
7555  
7560  
7565  
7570  
7575  
7580  
7585  
7590  
7595  
7600  
7605  
7610  
7615  
7620  
7625  
7630  
7635  
7640  
7645  
7650  
7655  
7660  
7665  
7670  
7675  
7680  
7685  
7690  
7695  
7700  
7705  
7710  
7715  
7720  
7725  
7730  
7735  
7740  
7745  
7750  
7755  
7760  
7765  
7770  
7775  
7780  
7785  
7790  
7795  
7800  
7805  
7810  
7815  
7820  
7825  
7830  
7835  
7840  
7845  
7850  
7855  
7860  
7865  
7870  
7875  
7880  
7885  
7890  
7895  
7900  
7905  
7910  
7915  
7920  
7925  
7930  
7935  
7940  
7945  
7950  
7955  
7960  
7965  
7970  
7975  
7980  
7985  
7990  
7995  
8000  
8005  
8010  
8015  
8020  
8025  
8030  
8035  
8040  
8045  
8050  
8055  
8060  
8065  
8070  
8075  
8080  
8085  
8090  
8095  
8100  
8105  
8110  
8115  
8120  
8125  
8130  
8135  
8140  
8145  
8150  
8155  
8160  
8165  
8170  
8175  
8180  
8185  
8190  
8195  
8200  
8205  
8210  
8215  
8220  
8225  
8230  
8235  
8240  
8245  
8250  
8255  
8260  
8265  
8270  
8275  
8280  
8285  
8290  
8295  
8300  
8305  
8310  
8315  
8320  
8325  
8330  
8335  
8340  
8345  
8350  
8355  
8360  
8365  
8370  
8375  
8380  
8385  
8390  
8395  
8400  
8405  
8410  
8415  
8420  
8425  
8430  
8435  
8440  
8445  
8450  
8455  
8460  
8465  
8470  
8475  
8480  
8485  
8490  
8495  
8500  
8505  
8510  
8515  
8520  
8525  
8530  
8535  
8540  
8545  
8550  
8555  
8560  
8565  
8570  
8575  
8580  
8585  
8590  
8595  
8600  
8605  
8610  
8615  
8620  
8625  
8630  
8635  
8640  
8645  
8650  
8655  
8660  
8665  
8670  
8675  
8680  
8685  
8690  
8695  
8700  
8705  
8710  
8715  
8720  
8725  
8730  
8735  
8740  
8745  
8750  
8755  
8760  
8765  
8770  
8775  
8780  
8785  
8790  
8795  
8800  
8805  
8810  
8815  
8820  
8825  
8830  
8835  
8840  
8845  
8850  
8855  
8860  
8865  
8870  
8875  
8880  
8885  
8890  
8895  
8900  
8905  
8910  
8915  
8920  
8925  
8930  
8935  
8940  
8945  
8950  
8955  
8960  
8965  
8970  
8975  
8980  
8985  
8990  
8995  
9000  
9005  
9010  
9015  
9020  
9025  
9030  
9035  
9040  
9045  
9050  
9055  
9060  
9065  
9070  
9075  
9080  
9085  
9090  
9095  
9100  
9105  
9110  
9115  
9120  
9125  
9130  
9135  
9140  
9145  
9150  
9155  
9160  
9165  
9170  
9175  
9180  
9185  
9190  
9195  
9200  
9205  
9210  
9215  
9220  
9225  
9230  
9235  
9240  
9245  
9250  
9255  
9260  
9265  
9270  
9275  
9280  
9285  
9290  
9295  
9300  
9305  
9310  
9315  
9320  
9325  
9330  
9335  
9340  
9345  
9350  
9355  
9360  
9365  
9370  
9375  
9380  
9385  
9390  
9395  
9400  
9405  
9410  
9415  
9420  
9425  
9430  
9435  
9440  
9445  
9450  
9455  
9460  
9465  
9470  
9475  
9480  
9485  
9490  
9495  
9500  
9505  
9510  
9515  
9520  
9525  
9530  
9535  
9540  
9545  
9550  
9555  
9560  
9565  
9570  
9575  
9580  
9585  
9590  
9595  
9600  
9605  
9610  
9615  
9620  
9625  
9630  
9635  
9640  
9645  
9650  
9655  
9660  
9665  
9670  
9675  
9680  
9685  
9690  
9695  
9700  
9705  
9710  
9715  
9720  
9725  
9730  
9735  
9740  
9745  
9750  
9755  
9760  
9765  
9770  
9775  
9780  
9785  
9790  
9795  
9800  
9805  
9810  
9815  
9820  
9825  
9830  
9835  
9840  
9845  
9850  
9855  
9860  
9865  
9870  
9875  
9880  
9885  
9890  
9895  
9900  
9905  
9910  
9915  
9920  
9925  
9930  
9935  
9940  
9945  
9950  
9955  
9960  
9965  
9970  
9975  
9980  
9985  
9990  
9995  
10000  
10005  
10010  
10015  
10020  
10025  
10030  
10035  
10040  
10045  
10050  
10055  
10060  
10065  
10070  
10075  
10080  
10085  
10090  
10095  
10100  
10105  
10110  
10115  
10120  
10125  
10130  
10135  
10140  
10145  
10150  
10155  
10160  
10165  
10170  
10175  
10180  
10185  
10190  
10195  
10200  
10205  
10210  
10215  
10220

10 [1] a roll-off reducing agent comprising one or more compounds selected  
from the group consisting of carboxylic acids having 2 to 20 carbon atoms  
having either OH group or groups or SH group or groups, monocarboxylic acids  
having 1 to 20 carbon atoms, and dicarboxylic acids having 2 to 3 carbon atoms,  
5 and salts thereof;

15 [2] a roll-off reducing agent composition comprising:  
a roll-off reducing agent comprising one or more compounds selected  
from the group consisting of carboxylic acids having 2 to 20 carbon atoms  
having either OH group or groups or SH group or groups, monocarboxylic acids  
having 1 to 20 carbon atoms, and dicarboxylic acids having 2 to 3 carbon atoms,  
and salts thereof;

20 an abrasive; and

25 water;

30 [3] a polishing composition comprising:  
water;  
an abrasive;  
a roll-off reducing agent comprising one or more compounds selected  
from the group consisting of carboxylic acids having 2 to 20 carbon atoms  
having either OH group or groups or SH group or groups, monocarboxylic acids  
having 1 to 20 carbon atoms, and dicarboxylic acids having 2 to 3 carbon atoms,  
and salts thereof; and

35 an intermediate alumina;

40 [4] a polishing composition comprising:  
(A) one or more compounds selected from carboxylic acids having 2 to  
20 carbon atoms having either OH group or groups or SH group or groups,

TO/2010-042701  
10  
15  
20  
25

monocarboxylic acids having 1 to 20 carbon atoms, and dicarboxylic acids having 2 to 3 carbon atoms, and salts thereof;

(B) one or more compounds selected from polycarboxylic acids having 4 or more carbon atoms and having neither OH group or groups nor SH group or groups, aminopolycarboxylic acids, amino acids and salts thereof; and

(C) one or more compounds selected from an intermediate alumina and an alumina sol;

an abrasive; and

water;

[5] a polishing composition comprising:

(A) one or more compounds selected from carboxylic acids having 2 to 20 carbon atoms having either OH group or groups or SH group or groups, monocarboxylic acids having 1 to 20 carbon atoms, and dicarboxylic acids having 2 to 3 carbon atoms, and salts thereof; and

(B) one or more compounds selected from polycarboxylic acids having 4 or more carbon atoms and having neither OH group or groups nor SH group or groups, aminopolycarboxylic acids, amino acids and salts thereof;

an abrasive; and

water;

[6] a process of reducing roll-off of a polished substrate, comprising applying to a substrate to be polished a roll-off reducing agent comprising one or more compounds selected from the group consisting of carboxylic acids having 2 to 20 carbon atoms having either OH group or groups or SH group or groups, monocarboxylic acids having 1 to 20 carbon atoms, and dicarboxylic acids having 2 to 3 carbon atoms, and salts thereof;

DRAFT-04-2016-060

[7] a process for producing a polished substrate, comprising a step of applying to a substrate to be polished a roll-off reducing agent comprising one or more compounds selected from the group consisting of carboxylic acids having 2 to 20 carbon atoms having either OH group or groups or SH group or groups, monocarboxylic acids having 1 to 20 carbon atoms, and dicarboxylic acids having 2 to 3 carbon atoms, and salts thereof;

5 [8] a polishing process of a substrate to be polished comprising a step of polishing the substrate to be polished with the polishing composition of any one of items [3] to [5] above; and

[9] a process for producing a substrate comprising a step of polishing a substrate to be polished with the polishing composition of any one of items [3] to [5] above.

#### BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a graph showing a roll-off in connection with the detection curve.

#### DETAILED DESCRIPTION OF THE INVENTION

##### 1. Roll-Off Reducing Agent

20 The roll-off reducing agent used in the present invention is one or more compounds selected from the group consisting of carboxylic acids having 2 to 20 carbon atoms having either OH group or groups or SH group or groups, monocarboxylic acids having 1 to 20 carbon atoms, and dicarboxylic acids having 2 to 3 carbon atoms, and salts thereof. Among them, the carboxylic acids having 2 to 20 carbon atoms having either OH group or groups or SH group or

groups, the dicarboxylic acids having 2 to 3 carbon atoms, and salts thereof are preferable, from the viewpoint of an effect of improving roll-off.

The carboxylic acid having 2 to 20 carbon atoms having either OH group or groups or SH group or groups includes oxycarboxylic acids, and compounds in which an oxygen atom of the OH group of the oxycarboxylic acid is substituted by a sulfur atom. It is desired that the number of carbon atoms of these carboxylic acids is from 2 to 20, preferably from 2 to 12, more preferably from 2 to 8, still more preferably from 2 to 6, from the viewpoint of the solubility to water. In addition, as oxycarboxylic acids, those having a hydroxyl group at  $\alpha$ -position of a carboxyl group are preferable, from the viewpoint of reducing roll-off.

It is desired that the number of carbon atoms of the monocarboxylic acid is from 1 to 20, preferably from 1 to 12, more preferably from 1 to 8, still more preferably from 1 to 6, from the viewpoint of the solubility to water.

The dicarboxylic acid is those having 2 to 3 carbon atoms, namely oxalic acid and malonic acid, from the viewpoint of reducing roll-off. Among these roll-off reducing agents, the oxycarboxylic acids are preferable, from the viewpoint of increasing the polishing rate. In addition, the dicarboxylic acids are preferable, from the viewpoint of reducing roll-off.

Concrete examples of the carboxylic acid having 2 to 20 carbon atoms having either OH group or groups or SH group or groups include glycolic acid, mercaptosuccinic acid, thioglycolic acid, lactic acid,  $\beta$ -hydroxypropionic acid, malic acid, tartaric acid, citric acid, isocitric acid, allocitric acid, gluconic acid, glyoxylic acid, glyceric acid, mandelic acid, tropic acid, benzilic acid, salicylic acid, and the like. Concrete examples of the monocarboxylic acid include formic

10  
15  
20  
25  
TOKUYODI 69724860

acid, acetic acid, propionic acid, butyric acid, isobutyric acid, valeric acid, isovaleric acid, hexanoic acid, heptanoic acid, 2-methylhexanoic acid, octanoic acid, 2-ethylhexanoic acid, nonanoic acid, decanoic acid, lauric acid, and the like. Among them, acetic acid, oxalic acid, malonic acid, glycolic acid, lactic acid, 5 malic acid, tartaric acid, glyoxylic acid, citric acid and gluconic acid are preferable; oxalic acid, malonic acid, glycolic acid, lactic acid, malic acid, tartaric acid, glyoxylic acid, citric acid and gluconic acid are more preferable; and oxalic acid, malonic acid, glycolic acid, tartaric acid and glyoxylic acid are especially preferable.

In addition, when oxalic acid, malic acid, tartaric acid, citric acid or gluconic acid is used alone or in combination with other roll-off reducing agent, it is preferable because clogging of the abrasive grains and polishing grounds in the polishing pad can be reduced, so that the deterioration of the polishing properties such as polishing rate and surface qualities by using the polishing pad for a long period of time can be prevented. Also, a frequent pad washing would not be necessary, namely the distance between the pad dressings can be remarkably extended, so that the productivity is increased, thereby making it preferable also from the viewpoint of economic advantages. Among them, oxalic acid, tartaric acid and citric acid are preferable, and especially citric acid is preferable. Each of the monocarboxylic acid and the dicarboxylic acid used in the present invention is selected from carboxylic acids having neither OH group or groups nor SH group or groups.

The salts of these acids (namely, carboxylic acids having 2 to 20 carbon atoms having either OH group or groups or SH group or groups, monocarboxylic acids having 1 to 20 carbon atoms, and dicarboxylic acids having 2 to 3 carbon

202501069247624015

20

25

atoms) are not particularly limited. Concretely, there are included salts with a metal, ammonium, an alkylammonium, an organic amine, and the like. Concrete examples of the metal include metals belonging to Group 1A, 1B, 2A, 2B, 3A, 3B, 4A, 6A, 7A or 8 of the Periodic Table (long period form). Among these metals, from the viewpoint of reducing roll-off, those metals belonging to Group 1A, 3A, 3B, 7A or 8 of the Periodic Table are preferable, and those metals belonging to Group 1A, 3A or 3B of the Periodic Table are more preferable. Sodium and potassium belonging to Group 1A are most preferable.

Concrete examples of the alkylammonium include tetramethylammonium, tetraethylammonium, tetrabutylammonium, and the like.

Concrete examples of the organic amine include dimethylamine, trimethylamine, alkanolamines, and the like.

Among these salts, ammonium salts, sodium salts and potassium salts are especially preferable.

## 2. Roll-Off Reducing Agent Composition

The roll-off reducing agent of the present invention can be used by formulating the agent in a polishing liquid comprising an abrasive and water. The polishing composition obtained above is especially referred to “roll-off reducing agent composition” in the present specification. Specifically, the roll-off reducing agent composition of the present invention comprises the above-mentioned roll-off reducing agent, an abrasive and water.

The content of the roll-off reducing agent in the roll-off reducing agent composition is preferably 0.01% by weight or more, from the viewpoints of reducing roll-off and increasing the polishing rate, and the content of the roll-off

10 0424759-00424740

20

25

reducing agent is preferably 5% by weight or less, from the viewpoints of economic advantages and improving surface qualities. The content of the roll-off reducing agent is more preferably from 0.01 to 3% by weight, still more preferably from 0.01 to 2% by weight, most preferably from 0.02 to 1% by weight. Here, the roll-off reducing agent can be used alone or in admixture of two or more kinds.

As the abrasive used in the present invention, any abrasives generally employed for polishing can be used. The abrasive includes, for instance, metals; carbides of metals or metalloids, nitrides of metals or metalloids, oxides of metals or metalloids, borides of metals or metalloids, diamond, and the like. The metals or metalloids include those elements belonging to the Groups 2A, 2B, 3A, 3B, 4A, 4B, 5A, 6A, 7A or 8 of the Periodic Table (long period form). Concrete examples of the abrasive include  $\alpha$ -alumina particles, silicon carbide particles, diamond particles, magnesium oxide particles, zinc oxide particles, cerium oxide particles, zirconium oxide particles, colloidal silica particles, fumed silica particles, and the like. It is preferable to use these abrasives in admixture of two or more kinds, from the viewpoint of increasing the polishing rate. Among them,  $\alpha$ -alumina particles, cerium oxide particles, zirconium oxide particles, colloidal silica particles, fumed silica particles, and the like are more preferable, and  $\alpha$ -alumina particles are especially preferable.

The average primary particle size of the abrasive is preferably from 0.01 to 3  $\mu\text{m}$ , more preferably from 0.02 to 0.8  $\mu\text{m}$ , especially preferably from 0.05 to 0.5  $\mu\text{m}$ , from the viewpoint of increasing the polishing rate. Further, when the primary particles are aggregated to form a secondary particle, the average secondary particle size is preferably from 0.05 to 3  $\mu\text{m}$ , more preferably from 0.1

10  
15  
TUZEDO:6976801

20

25

to 1.5  $\mu\text{m}$ , especially preferably from 0.2 to 1.2  $\mu\text{m}$ , from the viewpoint of increasing the polishing rate in the same manner as above, and from the viewpoint of reducing the surface roughness of a polished object. The average primary particle size of the abrasive is obtained by subjecting the abrasive to an image analysis by observing with a scanning electron microscope (favorably from 3000 to 30000 times), and determining the particle size as a number-average particle size. In addition, the average secondary particle size can be determined as volume-average particle size by using a laser diffraction method.

5 The specific gravity of the abrasive is preferably from 2 to 6, more preferably from 2 to 5, from the viewpoints of the dispersibility, the feed ability to the polishing device and recovery and reuse.

10 The content of the abrasive is preferably from 1 to 40% by weight, more preferably from 2 to 30% by weight, still more preferably from 3 to 15% by weight, of the roll-off reducing agent composition, from the viewpoints of having economic advantages and making the surface roughness of a polished object small, thereby efficiently polishing the substrate.

15 Water in the roll-off reducing agent composition of the present invention is used as a medium, and the content of water is preferably from 50 to 98.99% by weight, more preferably 60 to 98% by weight, still more preferably from 70 to 95% by weight, from the viewpoint of efficiently polishing the object to be polished.

20 In addition, the roll-off reducing agent composition of the present invention can contain other components as occasion demands.

25 As other components, there are included organic acids and salts other than those listed as the roll-off reducing agent. The other components include, for

instance, organic acids such as polycarboxylic acids, aminopolycarboxylic acids and amino acids, and salts thereof, inorganic acids and salts thereof, oxidizing agents, thickeners, dispersants, anticorrosive agents, basic substances, surfactants, and the like. Concrete examples of the organic acids and salts thereof, inorganic acids and salts thereof, and the oxidizing agents are those listed in Japanese Patent Laid-Open No. Sho 62-25187, page 2, upper right column, lines 3 to 11; Japanese Patent Laid-Open No. Sho 63-251163, page 2, lower left column, lines 7 to 14; Japanese Patent Laid-Open No. Hei 1-205973, page 3, upper left column, line 11 to upper right column, line 2; Japanese Patent Laid-Open No. Hei 3-115383, page 2, lower right column, line 16 to page 3, upper left column, line 11; Japanese Patent Laid-Open No. Hei 4-108887, page 2, lower left column, lines 1 to 9; Japanese Patent Laid-Open No. Hei 4-275387, page 2, right column, line 27 to page 3, left column, line 12; Japanese Patent Laid-Open No. Hei 4-363385, page 2, right column, lines 21 to 30, the entire contents of which are incorporated herein by reference, and the like.

These other components may be used alone or in admixture of two or more kinds. In addition, the content of the other components is preferably from 0.05 to 20% by weight, more preferably from 0.05 to 10% by weight, still more preferably from 0.05 to 5% by weight, of the roll-off reducing agent composition, from the viewpoint of exhibiting the respective functions and from the viewpoint of economic advantages.

The concentration of each component of the above-mentioned roll-off reducing agent composition is a preferable concentration during polishing, and it may be a concentration during the preparation of the composition. The composition is usually prepared as a concentrate, and the concentrate is diluted

2024/06/28 15:53:28

20

25

upon use in many cases.

The roll-off reducing agent composition of the present invention can be prepared by adding one or more compounds selected from the group consisting of the carboxylic acids having 2 to 20 carbon atoms having either OH group or groups or SH group or groups, the monocarboxylic acids having 1 to 20 carbon atoms, and the dicarboxylic acids having 2 to 3 carbon atoms, and salts thereof, and various additives, as occasion demands in proper amounts to water and an abrasive, and mixing the components by a known process.

It is preferable that the pH of the roll-off reducing agent composition is appropriately adjusted depending upon the kinds and the required qualities and the like of the substrate to be polished. For instance, the pH of the roll-off reducing agent composition is preferably from 2 to 12, from the viewpoints of the cleanability of the substrate and the anti-corrosiveness of the working machine, and from the viewpoint of the safety of the operator. In addition, in a case where a substrate to be polished is a substrate for precision parts which is mainly made of a metal such as an Ni-P plated aluminum alloy substrate, the pH is preferably from 2 to 9, especially preferably from 3 to 8, from the viewpoints of increasing the polishing rate and improving the surface qualities. When the roll-off reducing agent composition is used for polishing a semiconductor wafer, a semiconductor element, or the like, especially for polishing a silicon substrate, a poly-silicon film, an SiO<sub>2</sub> film, or the like, the pH is preferably from 7 to 12, more preferably from 8 to 12, especially preferably from 9 to 11, from the viewpoints of increasing the polishing rate and improving the surface qualities. The pH can be adjusted by adding properly an inorganic acid such as nitric acid or sulfuric acid, an organic acid such as a polycarboxylic acid, an

10  
15  
20  
25

aminopolycarboxylic acid, or an amino acid, a metal salt or an ammonium salt thereof, or a basic substance such as aqueous ammonia, sodium hydroxide, potassium hydroxide or amine in a desired amount as occasion demands.

5       3.     Process of Reducing Roll-Off of Polished Substrate by Using Roll-Off Reducing Agent and Process for Producing Polished Substrate

The material for an object to be polished as representatively exemplified by the substrate to be polished used in the present invention includes, for instance, metals or metalloids such as silicon, aluminum, nickel, tungsten, copper, tantalum and titanium; alloys made of these metals as main components; glassy substances such as glass, glassy carbon and amorphous carbons; ceramic materials such as alumina, silicon dioxide, silicon nitride, tantalum nitride and titanium nitride; resins such as polyimide resins; and the like. Among them, it is preferable that an object to be polished is made of a metal such as aluminum, nickel, tungsten or copper, or made of an alloy containing these metals as the main components; or an object to be polished is a semiconductor substrate made of semiconductor elements containing these metals. Especially, in a case where the roll-off reducing agent of the present invention is used when polishing an aluminum alloy substrate plated with Ni-P, it is preferable because the extent of roll-off can be made small.

The shape for the object to be polished is not particularly limited. For instance, those having shapes containing planar portions such as discs, plates, slabs and prisms, or shapes containing curved portions such as lenses can be subjects for polishing with the roll-off reducing agent composition of the present invention. Among them, those having the disc-shaped objects are especially

10  
15  
10/24/01 6:57:21p60

preferable in polishing.

The roll-off reducing agent of the present invention can be favorably used in polishing the substrate for precision parts. For instance, the roll-off reducing agent is suitable for polishing substrates for precision parts such as substrates for magnetic recording media for magnetic discs, optical discs, opto-magnetic discs, and the like; photomask substrates, optical lenses, optical mirrors, optical prisms and semiconductor substrates. The polishing of a semiconductor substrate comprises the steps of polishing a silicon wafer (bare wafer), forming separation membrane for an embedding element, flattening an interlayer insulating film, forming an embedded metal line, and forming embedded capacitor, and the like. The roll-off reducing agent composition of the present invention is especially suitable for polishing a magnetic disc substrate.

In the process of reducing roll-off of the polished substrate using the roll-off reducing agent of the present invention, the extent of roll-off of the polished substrate can be remarkably reduced by polishing the substrate to be polished listed above with a polishing liquid comprising the roll-off reducing agent of the present invention, or the roll-off reducing agent composition of the present invention *per se* as a polishing liquid.

For instance, a substrate with reduced roll-off can be produced by clamping a substrate with polishing discs to which a polishing cloth made of nonwoven organic polymer fabric, is pasted; feeding a polishing liquid comprising the roll-off reducing agent of the present invention, or the roll-off reducing agent composition of the present invention to a polishing surface; and moving the polishing discs or the substrate, with applying a given pressure.

The roll-off generated in the polished substrate in the present invention

10  
15  
20  
25

can be evaluated by determining the shape of the end surface part by using, for instance, a tracer or optical profilometer, and numerically expressing the extent of how much more the end surface is grounded as compared to the central portion of the disc in accordance with the profile.

5 The method of numerically expressing roll-off is determined as follows.

As shown in Figure 1, three points, i.e. point A, point B and point C, are taken on the detection curve which are given distances away from the center of the disc, the detection curve meaning the shape of the end surface part of the polished substrate). Roll-off refers to a distance (D) between point B and a base line, wherein the base line is defined as a straight line connecting point A and point C. The term "having good roll-off" refers to a value of D more approximating 0. The roll-off value refers to a value obtained by dividing D by 1/2 of the amount of variation in the thickness of the disc before and after polishing. The roll-off value is preferably 0.2  $\mu\text{m}/\mu\text{m}$  or less, more preferably 0.15  $\mu\text{m}/\mu\text{m}$  or less, still more preferably 0.10  $\mu\text{m}/\mu\text{m}$  or less.

Here, the positions of point A, point B and point C may vary depending upon the size of the object to be determined. In general, it is preferable that point B is positioned at 0.5 mm away from the end of the disc on the line connecting the end part and the center of the disc, that the point C is positioned at 2.5 mm away from the end, and that that the point A is positioned at 4.5 mm away from the end. For instance, in the case of 3.5 inch disc, it is preferable that point A, point B and point C are respectively positioned at 43 mm, 47 mm and 45 mm away from the center of the disc.

In addition, in the polishing process of a substrate for precision parts or the like, by using the roll-off reducing agent of the present invention, there are

10  
15  
20  
25

advantages that not only the roll-off of the substrate can be remarkably reduced, but also that the polishing rate can be increased. Also, in a case where one or more kinds selected from oxalic acid, malic acid, tartaric acid, citric acid, gluconic acid, and salts thereof are used as the roll-off reducing agent, it is

5 preferable because the clogging of the abrasive grains and polishing grounds in the polishing pad can be reduced, so that the deterioration of the polishing properties such as polishing rate and surface qualities by using the polishing pad for a long period of time can be prevented.

In this case, among the above-mentioned compounds, oxalic acid, tartaric acid, citric acid and salts thereof are preferable, and especially citric acid and salts thereof are preferable. In addition, in a case where the above-mentioned compounds are used in combination of two or more kinds, especially preferable combinations are combinations of two or more kinds selected from oxalic acid, tartaric acid, citric acid and salts thereof; or combinations of one or more kinds selected from oxalic acid, tartaric acid, citric acid and salts thereof, with one or more kinds selected from malonic acid, glycolic acid, lactic acid, malic acid, gluconic acid and salts thereof. More preferable are combinations of citric acid or a salt thereof with one or more kinds selected from oxalic acid, glycolic acid, lactic acid, malic acid, tartaric acid and salts thereof. Particularly preferable combinations are citric acid or a salt thereof with glycolic acid or a salt thereof.

20 The roll-off reducing agent composition of the present invention especially has an effect in the polishing process, and the roll-off reducing agent composition can be similarly applied to a process other than the polishing process, for instance, a lapping process, and the like.

4. Polishing Composition

The polishing composition of the present invention can be roughly classified into the following three embodiments:

[Embodiment 1] A polishing composition comprising:

5                   water;

an abrasive;

a roll-off reducing agent comprising one or more compounds selected from the group consisting of carboxylic acids having 2 to 20 carbon atoms having either OH group or groups or SH group or groups, monocarboxylic acids having 1 to 20 carbon atoms, and dicarboxylic acids having 2 to 3 carbon atoms, and salts thereof; and

an intermediate alumina.

[Embodiment 2] A polishing composition comprising:

(A) one or more compounds selected from carboxylic acids having 2 to 20 carbon atoms having either OH group or groups or SH group or groups, monocarboxylic acids having 1 to 20 carbon atoms, and dicarboxylic acids having 2 to 3 carbon atoms, and salts thereof;

(B) one or more compounds selected from polycarboxylic acids having 4 or more carbon atoms and having neither OH group or groups nor SH group or groups, aminopolycarboxylic acids, amino acids and salts thereof; and

(C) one or more compounds selected from an intermediate alumina and an alumina sol;

an abrasive; and

water.

25 [Embodiment 3] A polishing composition comprising:

10  
15  
20  
25

20 carbon atoms having either OH group or groups or SH group or groups,  
monocarboxylic acids having 1 to 20 carbon atoms, and dicarboxylic acids  
having 2 to 3 carbon atoms, and salts thereof; and

5 (B) one or more compounds selected from polycarboxylic acids having  
4 or more carbon atoms and having neither OH group or groups nor SH group or  
groups, aminopolycarboxylic acids, amino acids and salts thereof;  
an abrasive; and  
water.

10 Embodiment 1

15 The polishing composition of Embodiment 1, as described above,  
comprises water, an abrasive, a roll-off reducing agent, and an intermediate  
alumina.

20 As the abrasive usable in Embodiment 1, those abrasives which are  
generally used for polishing can be used. Examples of the abrasives are not  
particularly limited, as long as they are the same ones as those used in the above-  
mentioned roll-off reducing agent composition.

25 The content of the abrasive is preferably from 1 to 40% by weight, more  
preferably from 2 to 30% by weight, still more preferably from 3 to 15% by  
weight, of the polishing composition of Embodiment 1, from the viewpoints of  
having economic advantages and making the surface roughness of a polished  
object small, thereby making it possible to efficiently polish the substrate.

26 The roll-off reducing agent usable in Embodiment 1 may be the same  
ones as those mentioned above.

1042400-69224860

The content of the roll-off reducing agent is preferably from 0.01 to 5% by weight, more preferably from 0.015 to 4% by weight, still more preferably from 0.03 to 2% by weight, of the polishing composition of Embodiment 1, from the viewpoints of reducing roll-off and having economic advantages.

In addition, the intermediate alumina used in Embodiment 1 is a generic term referring to alumina particles other than  $\alpha$ -alumina particles. Concrete examples thereof include  $\gamma$ -alumina particles,  $\delta$ -alumina particles,  $\theta$ -alumina particles,  $\eta$ -alumina particles,  $\kappa$ -alumina particles, and mixtures thereof. Among them, the following intermediate aluminas are preferable, from the viewpoints of increase in the polishing rate and the effect of reducing the surface roughness of a polished object. The crystal forms of the intermediate alumina preferably include  $\gamma$ -alumina,  $\delta$ -alumina,  $\theta$ -alumina, and mixtures thereof, more preferably  $\gamma$ -alumina and  $\theta$ -alumina. In addition, the intermediate alumina has a specific surface area (BET method) of preferably from 30 to 300  $\text{m}^2/\text{g}$ , more preferably from 50 to 200  $\text{m}^2/\text{g}$ , and an average particle size of preferably from 0.01 to 5  $\mu\text{m}$ , more preferably from 0.05 to 5  $\mu\text{m}$ , still more preferably from 0.1 to 3  $\mu\text{m}$ , especially preferably from 0.1 to 1.5  $\mu\text{m}$ . The average particle size can be determined as a volume-average particle size by using a laser diffraction method (for instance, one commercially available from Horiba, LTD. under the trade name of LA-920). In addition, the content of each of the alkali metal and the alkaline earth metal in the intermediate alumina particles is preferably 0.1% by weight or less, more preferably 0.05% by weight or less, especially preferably 0.01% by weight or less.

For instance, in a case where aluminum hydroxide, an alumina sol or the like which has a relatively large specific surface area and a low content of the

10  
15  
20  
25

alkali metal and the alkaline earth metal is used as a raw material, since there is little fusion of the intermediate alumina produced and the particle strength is small, no surface defects are caused on a polished substrate, thereby making it especially effective in reducing the surface roughness of a polished object.

As the raw material which can be used in the preparation of the intermediate alumina, there can be used, for instance, aluminum hydroxide, alumina sols, and the like, which can be represented by the formulas  $\text{Al(OH)}_3$ ,  $\text{Al}_2\text{O}_3 \bullet 3\text{H}_2\text{O}$ ,  $\text{AlOOH}$ ,  $\text{Al}_2\text{O}_3 \bullet \text{H}_2\text{O}$ , and  $\text{Al}_2\text{O}_3 \bullet n\text{H}_2\text{O}$ , wherein n is a number of 1 to 3. The specific surface area of the raw material is preferably  $10 \text{ m}^2/\text{g}$  or more, more preferably  $30 \text{ m}^2/\text{g}$  or more, especially preferably  $50 \text{ m}^2/\text{g}$  or more. In addition, the content of each of the alkali metal and the alkaline earth metal in the raw material is preferably 0.1% by weight or less, more preferably 0.05% by weight or less, especially preferably 0.03% by weight or less. Further, in a case where an intermediate alumina is prepared by thermally dehydrating aluminum hydroxide, a forcible introduction of a dry air or nitrogen gas during baking is further effective in the reduction of surface defects and surface roughness of the polished substrate. Here, the above-mentioned thermal dehydration treatment can be carried out by a conventional method.

These intermediate aluminas are adjusted to a given particle size by wet pulverization or dry pulverization by using a pulverizer such as a ball-mill, a beads-mill, a high-pressure homogenizer or a jet mill as occasion demands.

Since the intermediate alumina is used together with the abrasive and the roll-off reducing agent mentioned above, the polishing rate is increased and surface defects such as pits are prevented, and the reduction in the surface roughness of the polished substrate can be further accelerated.

09842769.042704  
10  
15

It is desired that the content of the intermediate alumina in the polishing composition of Embodiment 1 is from 1 to 100 parts by weight, preferably from 2 to 70 parts by weight, more preferably from 4 to 40 parts by weight, based on 100 parts by weight of the abrasive, from the viewpoints of economic advantages, 5 an accelerated effect of polishing, and an effect of reducing the surface roughness of the polished substrate, and from the viewpoint of obtaining capability of preventing surface defects such as pits.

Water in the polishing composition of Embodiment 1 is used as a medium, and the content of water is preferably from 40 to 98% by weight, more preferably from 50 to 97% by weight, especially preferably from 60 to 95% by weight, from the viewpoint of being capable of efficiently polishing the object to be polished.

#### Embodiment 2

The polishing composition of Embodiment 2, as described above, comprises one or more compounds selected from Compounds (A), one or more compounds selected from Compounds (B), one or more compounds selected from Compounds (C), an abrasive, and water.

Compounds (A) usable in Embodiment 2 are the same as the roll-off 20 reducing agent usable in Embodiment 1 mentioned above.

The content of Compounds (A) is preferably from 0.01 to 5% by weight, more preferably from 0.015 to 3% by weight, still more preferably from 0.03 to 2% by weight, of the polishing composition of Embodiment 2, from the viewpoint of improving roll-off and from the viewpoint of having economic 25 advantages.

10  
15  
20  
25  
30  
35  
40  
45  
50  
55  
60  
65  
70  
75  
80  
85  
90  
95  
100

Compounds (B) usable in Embodiment 2 have an action of increasing the polishing rate. Compounds (B) include polycarboxylic acids having 4 or more carbon atoms and having neither OH group or groups nor SH group or groups, aminopolycarboxylic acids, amino acids and salts thereof.

Among the polycarboxylic acids having 4 or more carbon atoms and having neither OH group nor groups or SH group or groups, those having 4 to 20 carbon atoms are preferable, more preferably 4 to 10 carbon atoms, from the viewpoint of increasing the polishing rate. Also, for the same viewpoint as above, the aminopolycarboxylic acids preferably have a number of amino groups in one molecule of from 1 to 6, more preferably from 1 to 4, a number of carboxyl groups in one molecule of preferably from 1 to 12, more preferably 2 to 8, and a number of carbon atoms of preferably from 1 to 30, preferably from 1 to 20. For the same viewpoint as above, the amino acids preferably have a number of carbon atoms of preferably from 2 to 20, preferably from 2 to 10. Among them, polycarboxylic acids having 4 or more carbon atoms and having neither OH group or groups nor SH group or groups, aminopolycarboxylic acids, and salts thereof are preferable, from the viewpoint of increasing the polishing rate.

Concrete examples thereof include succinic acid, maleic acid, fumaric acid, glutaric acid, citraconic acid, itaconic acid, tricarballylic acid, adipic acid, propane-1,1,2,3-tetracarboxylic acid, butane-1,2,3,4-tetracarboxylic acid, diglycolic acid, nitrilotriacetic acid, ethylenediaminetetraacetic acid (EDTA), diethylenetriaminepentaacetic acid (DTPA), hydroxyethylenetetraacetic acid (HEDTA), triethylenetetraminehexaacetic acid (TTHA), dicarboxymethylglutamic acid (GLDA), glycine, alanine, and the like.

10  
15  
20  
25

Among them, succinic acid, maleic acid, fumaric acid, glutaric acid, citraconic acid, itaconic acid, tricarballylic acid, adipic acid, diglycolic acid, nitrilotriacetic acid, ethylenediaminetetraacetic acid and diethylenetriaminepentaacetic acid are preferable, and succinic acid, maleic acid, fumaric acid, citraconic acid, itaconic acid, tricarballylic acid, diglycolic acid, ethylenediaminetetraacetic acid and diethylenetriaminepentaacetic acid are more preferable.

In addition, the salts of these acids, i.e. salts of polycarboxylic acids having 4 or more carbon atoms and having neither OH group or groups nor SH group or groups, salts of aminopolycarboxylic acids and salts of amino acids, are not particularly limited. Concretely, there are included salts with a metal, ammonium, an alkylammonium, an organic amine, and the like. Concrete examples of the metal include metals belonging to Group 1A, 1B, 2A, 2B, 3A, 3B, 4A, 6A, 7A or 8 of the Periodic Table (long period form). Among these metals, from the viewpoint of increasing the polishing rate, the metals belonging to Group 1A, 3A, 3B, 7A or 8 of the Periodic Table are preferable, and the metals belonging to Group 1A, 3A, 3B or 8 of the Periodic Table are more preferable. Sodium and potassium belonging to Group 1A, cerium belonging to Group 3A, aluminum belonging to Group 3B and iron belonging to Group 8 are most preferable.

Concrete examples of the alkylammonium include tetramethylammonium, tetraethylammonium, tetrabutylammonium, and the like.

Concrete examples of the organic amine include dimethylamine, trimethylamine, alkanolamines, and the like.

Among these salts, ammonium salts, sodium salts, potassium salts and

10 T072403042769734860  
15

20

aluminum salts are especially preferable.

These compounds of Compounds (B) can be used alone or in admixture of two or more kinds.

A total content of Compounds (B) is preferably from 0.01 to 10% by weight, more preferably from 0.02 to 7% by weight, still more preferably from 0.03 to 5% by weight, of the polishing composition of Embodiment 2, from the viewpoint of the effect of accelerating polishing, the viewpoint of economic advantages, and the viewpoint of improvement in the surface qualities.

Also, in Embodiment 2, more preferable combinations of Compounds (A) with Compounds (B) are combinations of one or more compounds selected from Compounds (A), acetic acid, oxalic acid, malonic acid, glycolic acid, lactic acid, malic acid, glyoxylic acid, tartaric acid, citric acid, gluconic acid, and salts thereof, with one or more compounds selected from Compounds (B), succinic acid, maleic acid, fumaric acid, glutaric acid, citraconic acid, itaconic acid, adipic acid, tricarballylic acid, diglycolic acid, nitrilotriacetic acid, ethylenediaminetetraacetic acid, diethylenetriaminepentaacetic acid and salts thereof, from the viewpoints of increasing the polishing rate and reducing roll-off. Still more preferable are combinations of one or more compounds selected from Compounds (A), oxalic acid, malonic acid, glycolic acid, lactic acid, malic acid, glyoxylic acid, tartaric acid, citric acid, gluconic acid, and salts thereof, with one or more compounds selected from Compounds (B), succinic acid, maleic acid, fumaric acid, citraconic acid, itaconic acid, tricarballylic acid, diglycolic acid, ethylenediaminetetraacetic acid, diethylenetriaminepentaacetic acid and salts thereof. Especially preferable are combinations of one or more compounds selected from Compounds (A), glycolic acid, oxalic acid, tartaric acid, citric acid,

10  
15  
20  
25

malonic acid, and salts thereof, with one or more compounds selected from Compounds (B), succinic acid, maleic acid, itaconic acid, fumaric acid, ethylenediaminetetraacetic acid, diethylenetriaminepentaacetic acid and salts thereof. In addition, in a case where one or more compounds selected from 5 oxalic acid, malic acid, tartaric acid, citric acid, gluconic acid and salts thereof are used as Compounds (A), it is preferable, because clogging of the abrasive grains and polishing grounds in the polishing pad can be reduced, so that the deterioration of the polishing properties such as polishing rate and surface qualities by using the polishing pad for a long period of time can be prevented.

In this case, among Compounds (A), oxalic acid, tartaric acid, citric acid, and salts thereof are preferable, and citric acid and salts thereof are especially preferable. In addition, in a case where two or more kinds of Compounds (A) are used in combination, especially preferable combinations are combinations of two or more kinds selected from oxalic acid, tartaric acid, citric acid and salts thereof; or combinations of one or more kinds selected from oxalic acid, tartaric acid, citric acid and salts thereof with one or more kinds selected from malonic acid, glycolic acid, lactic acid, malic acid, gluconic acid and salts thereof. More preferable combinations are combinations of citric acid or a salt thereof with one or more kinds selected from oxalic acid, glycolic acid, lactic acid, malic acid, tartaric acid and salts thereof. Especially preferable combinations are combinations of citric acid or a salt thereof with glycolic acid or a salt thereof.

Compounds (C) used in Embodiment 2 comprise an intermediate alumina and alumina sol. The intermediate alumina is a generic term referring to alumina particles other than  $\alpha$ -alumina particles. The intermediate alumina may be the same ones as those usable in Embodiment 1 mentioned above.

2000-09-22 15:40:24

20

25

In addition, the alumina sol refers to those which can be represented by the formulas  $\text{AlOOH}$ ,  $\text{AlOOH} \bullet n\text{H}_2\text{O}$ , wherein  $n$  is a number of 1 to 3, for instance,  $\text{Al}_2\text{O}_3 \bullet \text{H}_2\text{O}$  and the like. The crystal forms of the alumina sol include boehmite, pseudo-boehmite and amorphous. The alumina sol can be prepared by subjecting aluminum hydroxide, for instance, gibbsite, to a hydrothermal treatment at 250°C or so, or hydrolyzing an aluminum alcoholate. The alumina sol has an average particle size of preferably from 0.01 to 5  $\mu\text{m}$ , more preferably from 0.05 to 5  $\mu\text{m}$ , still more preferably from 0.1 to 3  $\mu\text{m}$ , especially preferably from 0.1 to 1.5  $\mu\text{m}$ . The average particle size can be determined as a volume-average particle size by using a laser diffraction method. The alumina sol has a specific surface area (BET method) of preferably from 30 to 300  $\text{m}^2/\text{g}$ , more preferably from 50 to 200  $\text{m}^2/\text{g}$ .

Since the intermediate alumina and the alumina sol in Compounds (C) are used together with Compounds (A) and Compounds (B), the effects that the polishing rate is increased and surface defects such as pits are prevented, and the reduction in the surface roughness of the polished object can be further accelerated. In this case, the intermediate alumina and the alumina sol in Compounds (C) can be used alone or in admixture. Especially, the intermediate alumina is more preferable, from the viewpoints of an increase in the polishing rate, an extent of the effect of preventing surface defects and the like, and an effect of reducing the surface roughness of a polished object.

A total content of Compounds (C) in the polishing composition of Embodiment 2 is preferably from 1 to 100 parts by weight, more preferably from 2 to 70 parts by weight, still more preferably from 4 to 40 parts by weight, based on 100 parts by weight of the abrasive, from the viewpoints of economic

010  
09882795 07/24/15  
15

advantages, the effect of accelerating polishing, the effect of reducing the surface roughness of a polished object, and from the viewpoint of obtaining the capability of preventing surface defects such as pits.

As the abrasive usable in Embodiment 2, those abrasives which are generally used for polishing can be used. The abrasives may be the same ones as those used in the above-mentioned roll-off reducing agent composition.

The content of the abrasive is preferably from 1 to 40% by weight, more preferably from 2 to 30% by weight, still more preferably from 3 to 15% by weight, of the polishing composition of Embodiment 2, from the viewpoints of having economic advantages and making the surface roughness of a polished object small, thereby making it possible to efficiently polish the object to be polished.

Water in the polishing composition of Embodiment 2 is used as a medium, and the content of water is preferably from 40 to 98% by weight, more preferably 50 to 97% by weight, especially preferably from 60 to 95% by weight, from the viewpoint of being capable of efficiently polishing the object to be polished.

### Embodiment 3

The polishing composition of Embodiment 3, as described above, comprises one or more compounds selected from Compounds (A), one or more compounds selected from Compounds (B), an abrasive, and water.

Compounds (A) usable in Embodiment 3 have an action of improving roll-off caused in the polished substrate, and are the same ones as those exemplified as Compounds (A) in Embodiment 2 mentioned above can be used.

100-042747-9 2769-042747-0

The content of Compounds (A) is preferably from 0.01 to 5% by weight, more preferably from 0.015 to 3% by weight, still more preferably from 0.03 to 2% by weight, of the polishing composition of Embodiment 3, from the viewpoints of improving roll-off and having economic advantages.

5 Compounds (B) usable in Embodiment 3 have an action of increasing the polishing rate, and the same ones as those Compounds (B) which are used in Embodiment 2 mentioned above can be used.

The content of Compounds (B) is preferably from 0.01 to 10% by weight, more preferably from 0.02 to 7% by weight, still more preferably from 0.03 to 5% by weight, of the polishing composition of Embodiment 3, from the viewpoint of the effect of accelerating polishing, the viewpoint of economic advantages, and the viewpoint of improvement in the surface qualities.

As to the combinations of Compounds (A) and Compounds (B), the same ones as those combinations which are mentioned in Embodiment 2 can be used.

As the abrasive usable in Embodiment 3, those abrasives which are generally used for polishing can be used. Examples of the abrasives may be the same ones as those used in the above-mentioned roll-off reducing agent composition.

20 The content of the abrasive is preferably from 1 to 40% by weight, more preferably from 2 to 30% by weight, still more preferably from 3 to 15% by weight, of the polishing composition of Embodiment 3, from the viewpoints of having economic advantages and making the surface roughness of a polished object small, thereby making it possible to efficiently polish the substrate.

25 Water in the polishing composition of Embodiment 3 is used as a medium, and the content of water is preferably from 40 to 98% by weight, more

10  
15  
20  
25

preferably from 50 to 97% by weight, especially preferably from 60 to 95% by weight, from the viewpoint of being capable of efficiently polishing the object to be polished.

The polishing composition of the present invention, as representatively exemplified by Embodiments 1 to 3 described above, can contain other components as occasion demands. As other components, there are included organic acids and salts other than the above-mentioned roll-off reducing agent. The other components include, for instance, organic acids such as polycarboxylic acids, aminopolycarboxylic acids and amino acids, and salts thereof, inorganic acids and salts thereof, oxidizing agents, thickeners, dispersants, anticorrosive agents, basic substances, surfactants, and the like. Concrete examples of the organic acids and salts thereof, inorganic acids and salts thereof, and the oxidizing agents may be the same ones used in the above-mentioned roll-off reducing agent composition.

These other components may be used alone or in admixture of two or more kinds. In addition, the content of the other components is preferably from 0.05 to 20% by weight, more preferably from 0.05 to 10% by weight, still more preferably from 0.05 to 5% by weight, of the polishing composition, from the viewpoint of increasing the polishing rate, from the viewpoint of exhibiting the respective functions and from the viewpoint of economic advantages.

The concentration of each component of the above-mentioned polishing composition is a preferable concentration during polishing, and it may be a concentration during the preparation of the composition. The composition is usually prepared as a concentrate, and the concentrate is diluted upon use in many cases.

10  
15  
20  
25

The process for preparing the polishing composition of the present invention includes the following:

The polishing composition of Embodiment 1 can be prepared by adding together in proper amounts an abrasive, a roll-off reducing agent, an intermediate alumina, and water, and various additives, as occasion demands, each of which is exemplified above, and mixing the components by a known process.

Also, the polishing composition of Embodiment 2 can be prepared by adding together in proper amounts Compounds (A), Compounds (B), Compounds (C), an abrasive, water, and various additives as occasion demands, each of which is exemplified above, and mixing the components by a known process.

In addition, the polishing composition of Embodiment 3 can be prepared by adding together in proper amounts Compounds (A), Compounds (B), an abrasive, water, and various additives as occasion demands, each of which is exemplified above, and mixing the components by a known process.

It is preferable that the pH of the polishing composition of the present invention is appropriately adjusted depending upon the kinds and the required qualities and the like of the object to be polished. For instance, the pH of the polishing composition is preferably from 2 to 12, from the viewpoints of the 20 cleanliness of the substrate and anti-corrosiveness of the working machine, and from the viewpoint of the safety of the operator. In addition, in a case where an object to be polished is a substrate for precision parts which is mainly made of a metal such as an Ni-P plated aluminum alloy substrate, the pH is more preferably from 2 to 9, especially preferably from 3 to 8, from the viewpoints of increasing 25 the polishing rate and improving the surface qualities. When the polishing

10 04/24/2015  
15 04/24/2015  
20 04/24/2015  
25 04/24/2015

composition is used for polishing a semiconductor wafer, a semiconductor element, or the like, especially polishing a silicon substrate, a poly-silicon film, an SiO<sub>2</sub> film, or the like, the pH is preferably from 7 to 12, more preferably from 8 to 12, especially preferably from 9 to 11, from the viewpoints of increasing the 5 polishing rate and improving the surface qualities. The pH can be adjusted by adding properly an inorganic acid such as nitric acid or sulfuric acid, an organic acid, or a basic substance such as aqueous ammonia, sodium hydroxide, potassium hydroxide in a desired amount as occasion demands.

10 5. Polishing Process of Substrate to Be Polished by Using Polishing  
Composition

15 The polishing process of a substrate to be polished of the present invention comprises polishing a substrate to be polished by using the polishing composition of the present invention, or preparing a polishing liquid by mixing each component so as to give the composition of the polishing composition of the present invention. Especially, the substrate for precision parts can be suitably produced.

20 The material for the object to be polished as representatively exemplified by the substrate to be polished used in the present invention may be the same ones as those to which the above-mentioned roll-off reducing agent composition is used. Especially, in a case where the polishing composition of the present invention is used when polishing an aluminum alloy substrate plated with Ni-P, it is preferable because the roll-off can be reduced, the polishing rate can be increased, and the surface roughness of a polished object can be reduced without 25 causing surface defects.

10/27/10 69724860

The shape for the object to be polished is not particularly limited, and may be the same ones as those for the above-mentioned roll-off reducing agent composition.

The polishing composition of the present invention can be favorably used in polishing the substrate for precision parts. For instance, the polishing composition is suitable for polishing substrates for magnetic recording media for magnetic discs, optical discs, opto-magnetic discs, and the like; photomask substrates, optical lenses, optical mirrors, optical prisms and semiconductor substrates. The polishing of the semiconductor substrates comprises the steps of polishing a silicon wafer (bare wafer), forming separation membrane for an embedding element, flattening an interlayer insulating film, forming an embedded metal line, and forming embedded capacitor, and the like. The polishing composition of the present invention is especially suitable for polishing a magnetic disc substrate. Among the magnetic disc substrates, aluminum magnetic disc substrates plated with Ni-P can be especially suitably used.

6. Process for Producing Polished Substrate by Using Polishing Composition

In addition, a process for producing a polished substrate by using the polishing composition of the present invention includes, for instance, a process comprising clamping a substrate to be polished with polishing discs to which a polishing cloth made of nonwoven organic polymer fabric, is pasted; feeding a polishing composition of the present invention to a polishing surface; and moving the polishing discs or the substrate, with applying a given pressure, thereby reducing roll-off and surface roughness of the polished object, to give a substrate without surface defects. Here, the roll-off generated in the polished

10  
15  
20  
25

substrate in the present invention can be evaluated in the same manner as described above.

As described above, by using the polishing composition of the present invention, a high-quality substrate with reduced surface roughness and reduced roll-off as well as increased polishing rate can be produced with high production efficiency without generating surface defects on the polished substrate. In addition, when a polishing composition comprising one or more compounds selected from oxalic acid, malic acid, tartaric acid, citric acid, gluconic acid and salts thereof is used, it is preferable because clogging of the abrasive grains and polishing grounds in the polishing pad can be reduced, so that the deterioration of the polishing properties such as polishing rate and surface qualities by using the polishing pad for a long period of time can be prevented.

In this case, among the above-mentioned compounds, oxalic acid, tartaric acid, citric acid and salts thereof are preferable, and especially citric acid and salts thereof are preferable. When these compounds are used in combination of two or more kinds, especially preferable combinations are combinations of two or more kinds selected from oxalic acid, tartaric acid, citric acid and salts thereof; or combinations of one or more kinds selected from oxalic acid, tartaric acid, citric acid and salts thereof with one or more kinds selected from malonic acid, glycolic acid, lactic acid, malic acid, gluconic acid and salts thereof. More preferable are combinations of citric acid or a salt thereof with one or more kinds selected from oxalic acid, glycolic acid, lactic acid, malic acid, tartaric acid and salts thereof. Especially preferable combinations are combinations of citric acid or a salt thereof with glycolic acid or a salt thereof.

The polishing composition of the present invention especially has an

10 0447289-042415  
15

effect in the polishing process, and the polishing composition can be similarly applied to a process other than the polishing process, for instance, a lapping process, and the like.

5

## EXAMPLES

### Examples I-1 to I-10 and Comparative Examples I-1 to I-5

There were mixed together 7 parts by weight of an abrasive [ $\alpha$ -alumina (purity: about 99.9%) having primary average particle size: 0.23  $\mu\text{m}$ , and secondary average particle size: 0.5  $\mu\text{m}$ ], a given amount of a roll-off reducing agent used in Examples or a compound used in Comparative Examples as listed in Table 1, and balance ion-exchanged water, with stirring. Here, the pH of each composition in Examples I-1 to I-10 and Comparative Examples I-2 to I-4 was adjusted to 4 with an aqueous ammonia, and the pH of each composition in Comparative Examples I-1 and I-5 was adjusted to 4 with nitric acid, to give 100 parts by weight of each roll-off reducing agent composition of Examples I-1 to I-10 and Comparative Examples I-1 to I-5.

Using each of the resulting roll-off reducing agent compositions, a substrate surface made of an Ni-P plated aluminum alloy, the substrate surface having an average deviation, of all points from plane fit to test part surface Ra of 0.2  $\mu\text{m}$ , as determined by Talystep commercially available from Rank Taylor-Hobson Limited (size of tip end of profilometer: 25  $\mu\text{m} \times 25 \mu\text{m}$ , by-pass filter: 80  $\mu\text{m}$ , measurement length: 0.64 mm), a thickness of 0.8 mm and a diameter of 95 mm was polished with a double-sided processing machine under Set Conditions I for Double-Sided Processing Machine given below, to give a polished Ni-P plated, aluminum alloy substrate usable for magnetic recording.

10  
15  
20  
25

media.

Set Conditions I for Double-Sided Processing Machine are as follows.

5

Set Conditions I for Double-Sided Processing Machine

Double-sided processing machine: double-sided processing machine, Model 9B, manufactured by SPEEDFAM CO., LTD.

Processing pressure: 9.8 kPa

Polishing Pad: "POLYTEX DG-H" (manufactured by Rodel Nitta K.K.).

Disc rotational speed: 50 r/min

Feeding flow rate for a polishing composition: 100 ml/min

Polishing time period: 5 minutes

Number of substrate introduced: 10

After polishing, the value of roll-off generated in the polished substrate was determined by the method described below, and expressed as a relative value on the basis of the value of Comparative Example I-2. Also, the thickness of an Ni-P plated aluminum alloy substrate of Examples was determined by using a thickness tester (a laser thickness tester, commercially available from 20 Mitsutoyo Corporation, Model LGH-110/LHC-11N). A rate of decrease in the thickness was obtained from the changes in the thickness of the aluminum alloy substrate before and after polishing, and expressed as a relative value (relative polishing rate) on the basis of the polishing rate of Comparative Example I-1.

The results are shown in Table 1.

25

20220409Z24360

Method for Determination of Roll-Off

Determination device: Mitsutoyo form tracer SV-C624

Tip end radius of profilometer: 2  $\mu\text{m}$  (Code No. 178-381)

Pressure at profilometer: 0.7 mN or less

5 Speed: 0.2 mm/s

Analyzing software: SV-600 Fine Profile Analysis System, Version 1.01

Filter: LPF (Gaussian) 0.800 mm

Using the device as specified above, the shape of the end part of the disc at 42.5 mm to 47.5 mm away from the center of the disc was determined, and D was obtained by using an analyzing software in accordance with the above determination method by taking points A, B and C at 43 mm away from the center of the disc, at 47 mm away from the center, and at 45 mm away from the center, respectively. The value obtained by dividing D by 1/2 the amount of change of thickness of the disc before and after polishing is defined as the roll-off value.

2024/01/09 22:18:50

Table 1

Ex. No.	Added Compound	Amount (Parts by Weight)	Roll-Off (Relative Value)	Polishing Rate (Relative Value)
I-1	Lactic Acid	0.81	0.54	1.6
I-2	Glycolic Acid	0.69	0.23	1.4
I-3	Tartaric Acid	0.68	0.24	1.3
I-4	Citric Acid	0.58	0.41	1.5
I-5	Malic Acid	0.61	0.43	1.3
I-6	Glyoxylic Acid	0.83	0.34	1.3
I-7	Oxalic Acid	0.29	0.14	1.1
I-8	Malonic Acid	0.47	0.15	1.1
I-9	Acetic Acid	0.54	0.61	1.2
I-10	Glycolic Acid Citric Acid	0.69 0.10	0.21	1.4
<u>Comp. Ex. No.</u>				
I-1	None	—	Undetermin- able #1	1.0
I-2	Aluminum Nitrate	0.60	1.0 #2	—
I-3	Succinic Acid	0.53	0.83	—
I-4	Aluminumammonium Ethylenediamine- tetraacetate	0.83	1.54	—
I-5	Glycine	0.68	0.93	—

#1: Ski jump was caused, so that the roll-off was undeterminable.

#2: The roll-off value was 0.31  $\mu\text{m}/\mu\text{m}$ .

It is clear from the results in Table 1 that all of the roll-off reducing agent compositions obtained in Examples I-1 to I-10 have remarkably reduced roll-off, as compared to the roll-off reducing agent composition obtained in Comparative Examples I-1 to I-5. Also, it is also found that the roll-off reducing agent compositions of Examples I-1 to I-10 each of which comprises the roll-off reducing agent of the present invention have increased polishing rates, as compared with that of Comparative Example I-1.

Further, the roll-off reducing agent composition prepared in each of Example I-4, Example I-10 and Comparative Example I-3 was subjected to polishing evaluation mentioned above for 20 repeated times, and a ratio of a twentieth relative polishing rate to the first relative polishing rate was determined as a measure for an ability of preventing pad clogging. As a result, the ratio of a twentieth relative polishing rate to the first relative polishing rate in the roll-off reducing agent composition of Example I-4 was 0.97, the ratio in the roll-off reducing agent composition of Example I-10 was 0.95, and the ratio in the composition of Comparative Example I-3 was 0.62.

It is clear from the evaluation results of ability of preventing pad clogging for Examples I-4 and I-10 and Comparative Example I-3 that Examples I-4 and I-10 exhibit little deterioration in the polishing rate as compared to that in Comparative Example I-3, thereby exhibiting an excellent ability for preventing pad clogging.

### Preparation Example II-1 for Intermediate Alumina

An alumina vessel (200 mm in length  $\times$  100 mm in width  $\times$  100 mm in

height) was charged with 100 g of pseudo-boehmite particles having an average particle size of 25  $\mu\text{m}$ , a specific surface area of 250  $\text{m}^2/\text{g}$ , an alkali metal content of 0.003% by weight, and an alkaline earth metal content of 0.01% by weight. The pseudo-boehmite particles were heated in a muffle furnace at a heating rate of 50°C/minute, and baked at a baking temperature of 930°C for 4 hours with nitrogen gas stream at a flow rate of 5 L/minute, to give an intermediate alumina. The intermediate alumina was transferred to a 2-L alumina ball-mill, and ion-exchanged water was added thereto to prepare a 30% by weight slurry. Thereafter, alumina balls of 3 mm in diameters were introduced into the ball-mill to disrupt the slurry, to prepare intermediate alumina particles. The prepared intermediate alumina particles were found to have the crystal form of  $\gamma$ -alumina by analysis of X-ray diffraction peaks, and had an average particle size of 0.3  $\mu\text{m}$ , a specific surface area of 150  $\text{m}^2/\text{g}$ , an alkali metal content of 0.005% by weight, and an alkaline earth metal content of 0.01% by weight.

Examples II-1 to II-6 and Comparative Examples II-1 to II-5

There were mixed together an abrasive [ $\alpha$ -alumina (purity: about 99.9%) having primary average particle size: 0.25  $\mu\text{m}$ , and secondary average particle size: 0.8  $\mu\text{m}$ ], a roll-off reducing agent, an intermediate alumina ( $\gamma$ -alumina) obtained in Preparation Examples II-1, and balance ion-exchanged water, to give a composition as shown in Table 2 with stirring. Here, the pH of each composition in Examples II-1 to II-5 and Comparative Examples II-2, II-3 and II-5 was adjusted to 4.0 with an aqueous ammonia, and the pH of each composition in Comparative Examples II-1 and II-4 was adjusted to 4.0 with

nitric acid, to give 100 parts by weight of each polishing composition of Examples II-1 to II-6 and Comparative Examples II-1 to II-5.

Table 2

Example No.	$\alpha$ -Alumina (Parts by Weight)	Roll-Off Reducing Agent	Amount (Parts by Weight)	Intermediate Alumina or Alumina Sol <sup>1)</sup>	Amount (Parts by Weight)
II-1	7	Glycolic Acid	0.15	Intermediate Alumina	1
II-2	7	Malic Acid	0.15	Intermediate Alumina	1
II-3	7	Tartaric Acid	0.15	Intermediate Alumina	1
II-4	7	Malonic Acid	0.15	Intermediate Alumina	1
II-5	7	Citric Acid	0.15	Intermediate Alumina	1
II-6	7	Glycolic Acid	0.15	Intermediate Alumina	1
		Citric Acid	0.05		
<hr/>					
<u>Comparative Example No.</u>					
II-1	7	None	—	None	—
II-2	7	Diammonium Ethylenediamine-tetraacetate	0.15	Intermediate Alumina	1
II-3	7	Succinic Acid	0.15	Alumina Sol	1
II-4	7	Gluconic Acid	0.15	Alumina Sol	1
II-5	7	Glycolic Acid	0.15	Alumina Sol	1

1): Commercially available from Nissan Chemical Industries, Ltd, under the trade name of Alumina Sol-200

Using each of the resulting polishing compositions, a substrate surface made of an Ni-P plated aluminum alloy, the substrate surface having an average deviation, of all points from plane fit to test part surface Ra of 0.2  $\mu\text{m}$ , as determined by the following method, a thickness of 0.8 mm and a diameter of 95 mm was polished with a double-sided processing machine under Set Conditions II for Double-Sided Processing Machine given below, to give a polished Ni-P plated, aluminum alloy substrate usable for magnetic recording media.

Set Conditions II for Double-Sided Processing Machine are as follows.

Set Conditions II for Double-Sided Processing Machine

Double-sided processing machine: double-sided processing machine, Model 9B, manufactured by SPEEDFAM CO., LTD.

Processing pressure: 9.8 kPa

Polishing Pad: "POLYTEX DG-H" (manufactured by Rodel Nitta K.K.).

Disc rotational speed: 55 r/min

10 Feeding flow rate for a polishing composition: 100 mL/min

Polishing time period: 4 minutes

Number of substrate introduced: 10

15 After polishing, the thickness of an Ni-P plated aluminum alloy substrate of Examples was determined by using a thickness tester (a laser thickness tester, commercially available from Mitsutoyo Corporation, Model LGH-110/LHC-11N). A rate of decrease in the thickness was obtained from the changes in the

thickness of the aluminum alloy substrate before and after polishing, and expressed as a relative value (relative polishing rate) on the basis of the polishing rate of Comparative Example II-1.

In addition, the surface roughness (average deviation, of all points from plane fit to test part surface Ra) and pits (surface defects) of each substrate after polishing were determined in accordance with the following methods, and roll-off was determined in the same manner as above. Here, the average deviation, of all points from plane fit to test part surface Ra was obtained as a relative value (relative roughness) on the basis of the surface roughness of Comparative Example II-1. In addition, the roll-off was obtained as a relative value (relative roll-off) on the basis of the roll-off value of Comparative Example II-2. The results are shown in Table 3.

[Average deviation of all points from plane fit to test part surface Ra]

Determined under the following conditions by using Talystep  
commercially available from Rank Taylor-Hobson Limited

Size of tip end of profilometer: 25  $\mu\text{m} \times 25 \mu\text{m}$

Bv-pass filter: 80  $\mu$ m

Measurement length: 0.64 mm

### [Surface defects (pits)]

The surface of each substrate was observed with an optical microscope (differential interference microscope) at a magnification of 200 times at an interval of 30° for 12 locations, and the number of pits was counted and evaluated as follows:

- S: 0  
 A: 1 to 3  
 B: 4 to 10  
 C: 10 or more

5

Table 3

Example No.	Polishing Rate (-)	Evaluation of Properties		
		Surface Roughness (-)	Surface Defects (Pits)	Roll-Off (-)
II-1	1.6	0.68	S	0.21
II-2	1.7	0.65	S	0.23
II-3	1.6	0.70	S	0.24
II-4	1.4	0.65	S	0.25
II-5	1.7	0.72	S	0.25
II-6	1.6	0.70	S	0.20

  

Comparative Example No.				
II-1	1	1	C	Undeter- minable <sup>*1</sup>
II-2	1.6	0.70	S	1 <sup>*2</sup>
II-3	1.5	1.2	A	0.83
II-4	1.4	1.1	A	0.40
II-5	1.1	1.1	A	0.25

\*1: Ski jump was caused, so that the roll-off was undeterminable.

\*2: The roll-off value was 0.36  $\mu\text{m}/\mu\text{m}$ .

It is clear from the results in Table 3 that all of the polishing compositions obtained in Examples II-1 to II-6 have high polishing rates, and especially as

compared with the polishing compositions obtained in Comparative Examples II-1 to II-5, the polishing compositions obtained in Examples II-1 to II-6 have reduced surface roughness, no surface defects, and markedly reduced roll-off in the polished substrate.

5

Further, the polishing composition prepared in each of Example II-5, Example II-6 and Comparative Example II-3 was subjected to polishing evaluation mentioned above for 20 repeated times, and a ratio of a twentieth relative polishing rate to the first relative polishing rate was determined as a measure for an ability of preventing pad clogging. As a result, the ratio of a twentieth relative polishing rate to the first relative polishing rate in the polishing composition of Example II-5 was 0.91, the ratio in the polishing composition of Example II-6 was 0.90, and the ratio in the polishing composition of Comparative Example II-3 was 0.50.

It is clear from the evaluation results of ability of preventing pad clogging for the polishing compositions of Examples II-5 and II-6 exhibit excellent ability for preventing pad clogging, as compared with that of Comparative Example II-3.

#### Preparation Example III-1 for Intermediate Alumina

An alumina vessel (200 mm in length × 100 mm in width × 100 mm in height) was charged with 100 g of pseudo-boehmite particles having an average particle size of 15  $\mu\text{m}$ , a specific surface area of  $240 \text{ m}^2/\text{g}$ , an alkali metal content of 0.002% by weight, and an alkaline earth metal content of 0.01% by weight. The pseudo-boehmite particles were heated in a muffle furnace at a heating rate of  $50^\circ\text{C}/\text{minute}$ , and baked at a baking temperature of  $900^\circ\text{C}$  for

10/24/2015 10:59:57 AM

15

20

25

4 hours with nitrogen gas stream at a flow rate of 5 L/minute, to give an intermediate alumina. The intermediate alumina was transferred to a 2-L alumina ball-mill, and ion-exchanged water was added thereto to prepare a 30% by weight slurry. Thereafter, alumina balls of 3 mm in diameters were  
5 introduced into the ball-mill to disrupt the slurry, to prepare intermediate alumina particles. The prepared intermediate alumina particles were found to have the crystal form of  $\gamma$ -alumina by analysis of X-ray diffraction peaks, and had an average particle size of 0.3  $\mu\text{m}$ , a specific surface area of 120  $\text{m}^2/\text{g}$ , an alkali metal content of 0.003% by weight, and an alkaline earth metal content of 0.01% by weight.

Examples III-1 to III-8 and Comparative Examples III-1 to III-5

There were mixed together an abrasive [ $\alpha$ -alumina (purity: about 99.9%) having primary average particle size: 0.25  $\mu\text{m}$ , and secondary average particle size: 0.65  $\mu\text{m}$ ], Compounds (A), Compounds (B), Compounds (C) [an intermediate alumina ( $\gamma$ -alumina) obtained in Preparation Examples III-1 or an alumina sol (boehmite)], and balance ion-exchanged water, to give a composition as shown in Table 4 with stirring. Here, the pH of each composition in Examples III-1 to III-8 and Comparative Examples III-1 to III-5 was adjusted to  
20 4.0 or 7.0 with nitric acid or an aqueous ammonia, to give 100 parts by weight of each polishing composition of Examples III-1 to III-8 and Comparative Examples III-1 to III-5.

098842769 • 042704  
10  
15

Table 4

Ex. No.	$\alpha$ -Alumina (parts by weight)	Compounds (A)		Compounds (B)		Compounds (C)		pH
		Compound	Amount (parts by weight)	Compound	Amount (parts by weight)	Compound	Amount (parts by weight)	
III-1	7	Glycolic acid	0.15	Aluminumm ammonium ethylene- diaminetetraacetate	0.8	Intermediate	1	4
III-2	7	Glycolic acid	0.15	Maleic acid	0.5	Alumina	1	7
III-3	7	Oxalic acid	0.15	Itaconic acid	0.5	Intermediate	1	7
III-4	7	Tartaric acid	0.15	Formic acid	0.5	Alumina	1	7
III-5	7	Citric acid	0.15	Succinic acid	0.5	Intermediate	1	7
III-6	7	Glycolic acid	0.15	Diammonium ethylenediamine tetraacetate	0.8	Alumina	1.5	4
III-7	7	Glycolic acid	0.15	Glycine	0.5	Intermediate	1	7
III-8	7	Glycolic acid Citric acid	0.05	Aluminumm ammonium ethylene- diaminetetraacetate	0.8	Intermediate	1	4

  

Comp. Ex. No.	None	None	None	Aluminumm ammonium ethylene- diaminetetraacetate	Aluminumm ammonium ethylene- diaminetetraacetate	None	None	None
III-1	7	None	—	—	—	—	—	—
III-2	7	None	—	Aluminumm ammonium ethylene- diaminetetraacetate	0.8	Intermediate	1	4
III-3	7	Glycolic acid	0.15	Aluminumm ammonium ethylene- diaminetetraacetate	0.8	Alumina	—	4
III-4	7	Glycolic acid	0.15	None	—	Intermediate	1	7
III-5	7	None	—	Diammonium ethylenediamine tetraacetate	0.8	Alumina	1.5	4

\*1. Commercially available from Nissan Chemical Industries, Ltd. under the trade name of Alumina Sol-200.

Using each of the resulting polishing compositions, a substrate surface made of an Ni-P plated aluminum alloy, the substrate surface having an average deviation, of all points from plane fit to test part surface Ra of 0.2  $\mu\text{m}$ , as determined by the method described above, a thickness of 0.8 mm and a 5 diameter of 95 mm was polished with a double-sided processing machine under Set Conditions II for Double-Sided Processing Machine given above, to give a polished Ni-P plated, aluminum alloy substrate usable for magnetic recording media.

After polishing, the thickness of the aluminum alloy substrate of Examples was determined by using a thickness tester (a laser thickness tester, commercially available from Mitsutoyo Corporation, Model LGH-110/LHC-11N). A rate of decrease in the thickness was obtained from the changes in the thickness of an Ni-P plated aluminum alloy substrate before and after polishing, and expressed as a relative value (relative polishing rate) of Comparative Example III-1.

In addition, the surface roughness (average deviation, of all points from plane fit to test part surface Ra), pits, and roll-off of each substrate after 20 polishing were determined in the same manner in accordance with the methods described above. Here, the average deviation, of all points from plane fit to test part surface Ra was obtained as a relative value (relative roughness) on the basis of the surface roughness of Comparative Example III-1. In addition, the roll-off was obtained as a relative value (relative roll-off) on the basis of the roll-off value of Comparative Example III-2. The results are shown in Table 5.

10  
09842769-072015  
15

Table 5

Ex. No.	Evaluation of Properties			
	Polishing Rate (-)	Surface Roughness (-)	Surface Defects (Pits)	Roll-Off (-)
III-1	2.3	0.70	S	0.26
III-2	2.5	0.75	S	0.28
III-3	2.3	0.67	S	0.30
III-4	2.4	0.71	S	0.31
III-5	2.3	0.68	S	0.31
III-6	1.8	0.95	A	0.31
III-7	2.0	0.83	S	0.42
III-8	2.4	0.74	S	0.25
<u>Comp. Ex. No.</u>				
III-1	1.0	1.0	C	Undeter- minable*
III-2	2.2	0.76	S	1.0
III-3	1.8	1.80	B	0.29
III-4	1.4	0.65	S	0.28
III-5	1.7	0.92	A	1.05

\*: Undeterminable due to generation of ski jump.

It is clear from the results in Table 5 that all of the polishing compositions obtained in Examples III-1 to III-8 are excellent, simultaneously satisfying all of effects of increasing polishing rates, effects of reducing surface defects such as pits, effects of reducing surface roughness and effects of reducing roll-off, as compared with those of the polishing compositions obtained in Comparative Examples III-1 to III-5.

Further, the polishing composition prepared in each of Example III-5, Example III-8 and Comparative Example III-2 was subjected to polishing evaluation mentioned above for 20 repeated times, and a ratio of a twentieth relative polishing rate to the first relative polishing rate was determined as a measure for an ability of preventing pad clogging. As a result, the ratio of a twentieth relative polishing rate to the first relative polishing rate in the polishing composition of Example III-5 was 0.91, the ratio in the polishing composition of Example III-8 was 0.90, and the ratio in the polishing composition of Comparative Example III-2 was 0.48.

It is clear from the evaluation results of ability of preventing pad clogging for the polishing compositions of Examples III-5 and III-8 exhibit excellent ability for preventing pad clogging, as compared with that of Comparative Example III-2.

#### Examples IV-1 to IV-7 and Comparative Examples IV-1 to IV-3

There were mixed together an abrasive [ $\alpha$ -alumina (purity: about 99.9%) having primary average particle size: 0.23  $\mu\text{m}$ , and secondary average particle size: 0.6  $\mu\text{m}$ ], Compounds (A), Compounds (B), and ion-exchanged water, and other components as occasion demands, to give a composition as shown in Table 6 with stirring. Here, the pH of each composition in Examples IV-1 to IV-7 and Comparative Examples IV-2 to IV-3 was adjusted with nitric acid, and the pH of the composition in Comparative Example IV-1 was adjusted with an aqueous ammonia, to a pH of 4.0 or 7.0, to give 100 parts by weight of each polishing composition of Examples IV-1 to IV-7 and Comparative Examples IV-1 to IV-3.

100-09724860  
10  
15

Using each of the resulting polishing compositions, a substrate surface made of an Ni-P plated aluminum alloy, the substrate surface having an average deviation, of all points from plane fit to test part surface Ra of 0.2  $\mu\text{m}$ , as determined by the method described above, a thickness of 0.8 mm and a diameter of 95 mm was polished with a double-sided processing machine under Set Conditions II for Double-Sided Processing Machine given above, to give a polished Ni-P plated, aluminum alloy substrate usable for magnetic recording media.

After polishing, the thickness of the aluminum alloy substrate of Examples was determined by using a thickness tester (a laser thickness tester, commercially available from Mitutoyo Corporation, Model LGH-110/LHC-11N). A rate of decrease in the thickness was obtained from the changes in the thickness of an Ni-P plated aluminum alloy substrate before and after polishing, and expressed as a relative value (relative polishing rate) of Comparative Example IV-1.

In addition, the surface roughness (average deviation, of all points from plane fit to test part surface Ra) and roll-off of each substrate after polishing were determined in the same manner in accordance with the methods described above. Here, the roll-off was obtained as a relative value (relative roll-off) on the basis of the roll-off value of Comparative Example IV-2. The results are shown in Table 6.

DRAFT 2759.012/2014

Table 6

Ex. No.	Compounds (A)		Compounds (B)		Others		pH Polishing Rate	Evaluation of Properties Roll-Off Rate
	$\alpha$ -Alumina (parts by weight)	Compound	Amount (parts by weight)	Compound	Amount (parts by weight)	Others (parts by weight)		
IV-1	7	Glycolic acid	0.15	Aluminumammonium- ethylenediamine- tetracetate	0.8	—	4	1.9 0.21
IV-2	7	Glycolic acid	0.15	Maleic acid	0.5	—	7	2.1 0.22
IV-3	7	Tartaric acid	0.15	Itaconic acid	0.5	—	7	1.9 0.24
IV-4	7	Malonic acid	0.15	Fumaric acid	0.5	—	7	1.9 0.27
IV-5	7	Citric acid	0.15	Diglycolic acid	0.5	—	7	2.0 0.24
IV-6	7	Glycolic acid	0.15	Diammonium- ethylenediamine- tetracetate	0.8	—	4	1.9 0.25
IV-7	7	Glycolic acid	0.15	Maleic acid	0.5	—	7	2.0 0.21
<u>Comparative Examples</u>								
IV-1	7	None	—	None	—	—	4	1 Undeter- minable <sup>1)</sup>
IV-2	7	None	—	Aluminumammonium- ethylenediamine- tetracetate	0.8	—	4	1.8 1 <sup>3)</sup>
IV-3	7	None	—	Succinic acid	0.6	Alumina Sol <sup>2)</sup> (0.4)	4	1.5 0.90

1) Undeterminable due to generation of ski jump.

2) Commercially available from Nissan Chemical Industries, Ltd. under the trade name of Alumina Sol-200.

3) The roll-off value was 0.37  $\mu\text{m}/\mu\text{m}$ .

It is clear from the results in Table 6 that all of the polishing compositions obtained in Examples IV-1 to IV-7 have high polishing rates, and markedly reduced roll-off in the polished substrate, as compared with the polishing compositions obtained in Comparative Examples IV-1 to IV-3.

5

Further, the polishing composition prepared in each of Example IV-5, Example IV-7 and Comparative Example IV-3 was subjected to polishing evaluation mentioned above for 20 repeated times, and a ratio of a twentieth relative polishing rate to the first relative polishing rate was determined as a measure for an ability of preventing pad clogging. As a result, the ratio of a twentieth relative polishing rate to the first relative polishing rate in the polishing composition of Example IV-5 was 0.95, the ratio in the polishing composition of Example IV-7 was 0.92, and the ratio in the polishing composition of Comparative Example IV-3 was 0.55.

It is clear from the evaluation results of ability of preventing pad clogging for the polishing compositions of Examples IV-5 and IV-7 exhibit excellent ability for preventing pad clogging, as compared with that of Comparative Example IV-3.

20

By polishing a substrate for precision parts or the like with the roll-off reducing agent of the present invention, there can be exhibited excellent effects that the roll-off of the polished substrate can be remarkably reduced, and that the polishing rate can be increased.

25

In addition, by polishing a substrate for precision parts or the like with the polishing composition of the present invention, there can be exhibited excellent

10  
09842769-042704  
15  
20

effects that the roll-off and the surface roughness of the polished substrate are remarkably reduced.

#### EQUIVALENTS

5 The present invention being thus described, it will be obvious that the same may be varied in many ways. Such variations are not to be regarded as a departure from the spirit and scope of the invention, and all such modifications as would be obvious to one skilled in the art are intended to be included within the scope of the following claims.

10  
15072400-002769724860